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All drawings by Milton Franklin Sonday, Jr. in collaboration with the authors.

PRINCIPLES OF TEXTILE CONSERVATION SCIENCE No. III

CLASSIFICATION OF FIBERS FOUND IN ANCIENT TEXTILES

JAMES W. RICE

(This particular paper is intended as introduction to technical discussions of dyes and dyeing, detergents, cleaning agents and processes and physical methods of conserving old and weak fabrics, all of which are in the course of preparation.)

1. What are the Systems of Describing the Natural Textile Fibers?

It is customary to classify the natural textile fibers into sources of origin such as animal, mineral and vegetable but conservators will find that a better way is to assign them into chemical divisions because it helps them to predict weaknesses and safe handling methods. This permits a scientific systematization of the necessary knowledge.

One of the three, the mineral, has very little immediate interest to the present day conservator and will not be discussed further. The several vegetable classes are assigned to the "cellulose" fiber category because they are composed principally of the chemical cellulose or a modified form called "ligno-cellulose." Of all the fibers we may encounter, cotton and well made paper are probably the most nearly pure cellulose. Cotton differs because it is a seed hair type of unique cellular structure. Other vegetable fiber substances made from the stalks and leaves of plants such as flax (linen), hemp, ramie, jute, wood, bark, and very many others are called bast fibers. Although both cellulosic fiber types contain some impurities such as waxes and minerals, the bast fibers are more apt to contain lignins and ligno-cellulose which are physically weaker and react differently with certain other substances than pure cellulose. A case in point is that ligno-cellulose is faintly acidic and for that reason tends to accept basic dyes, whereas true cellulose does not. Because the cellulose forms the bone structure of the plant in the bast fibers, the cellular form is apt to be long with channeled pores. This feature helps to explain differences in physical behavior between the bast and the seed hair type. The general chemical composition of cellulose which in the main governs the reactive behavior of this whole group of fibers was explained briefly in our first paper on *General Chemical and Physical Structural Features of the Natural Textile Fibers* and in it we find that cellulose resembles a sugar in composition and that it is not particularly active chemically. Because the cellulose molecule contains many ^{-}OH groups, it will attract molecules of water or $H^{-}OH$ by hydrogen bonding effects. This accounts for much of its water absorbtion and holding power.

The animal class of fibers is composed of proteins which are substances formed of amino-acids which among other elements contain nitrogen not found in pure cellulose. The proteins act chemically quite differently from cellulose and a knowledge of these differences should be of value to the conservator in suggesting how to handle this class of fibers.

2. What are Some of the Animal or Protein Fibers of Interest to the Conservator?

Protein fibers such as conservators may work with are: wool, and other animal hairs such as goat, camel, alpaca, and llama, and other substances such as feathers, leather, fur pelts and skins of various animals, silk, both domesticated and wild and perhaps sinew.

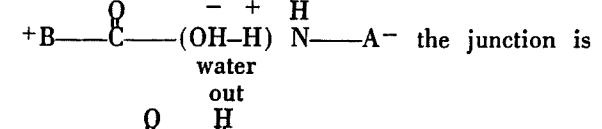
3. What are the Characteristics of the Protein Molecule Structure and How Does It Differ from the Cellulose Molecular Structure?

Like all fiber forming chemicals, the protein molecule is made up of long chains formed by polymerization of smaller molecules or radicals. The building blocks of the protein molecular chain are amino-acids. Many different amino-acids, however, are joined together in a regular sequence to form the final protein molecule. These amino-acids are amphoteric, which means that one end is acidic because of an acid carboxyl group $^{-}COOH$, while the other end is basic because of an amine group $^{-}NH_2$. The acid group ends can react chemically with the basic end of another amino-acid.

There are two ways in which this may occur. First, in natural animal growth processes, two amino-acids from digested foods and which are made soluble thereby come together at the growth site in the body and through the influence of a catalyst, probably an enzyme, join acid to base ends with the elimination of a molecule of water at the junction point. This is called a "peptide" linkage. This bond is a relatively strong chemical union but can be split by hydrolysis with pepsin or other body enzymes in the digestive processes. Strong alkalies also have damaging power on this connection.

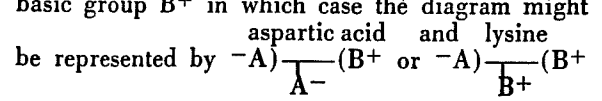
A second mode of interaction between the acid and base endings which is not as strong as the peptide linkage is the "salt bridge." This is an attractive force between the amine and carboxyl components of two or more parts in close proximity. This is one of the ways that molecules within a cell structure are held together. These acid and base sites are very important for another reason. They are able to combine with dyes that are either acid or basic and thus form relatively stable colored compounds. The "salt bridge" also attracts molecules of water, which is one of the causes of wet swelling of protein fibers.

Perhaps the following series of diagrams and structural formulas will augment and clarify the foregoing discussion. Let us take glycine as an example. It is probably the simplest of the fifteen amino-acids occurring in wool and is represented by chemists by the structural formula NH_2CH_2COOH in which ^-COOH is the carboxyl group or acid end and NH_2^- is the amine group or basic end. If we draw a simple diagram in which A^- stands for acid group, B^+ stands for basic group and a line stands for a series of carbon and other atoms extending between, we can represent the amino-acid like this ^-A——B^+ and where two of these form a peptide linkage with elimination of water thus

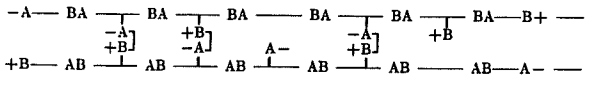


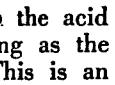
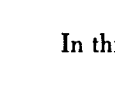
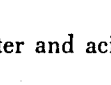
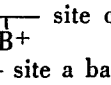
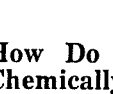
the junction is written (—C——N——) we will have our diagram show ——(AB)—— as the peptide linkage where + and — are neutralized.

Some of the more complex amino-acids contain more than one acid group A^- and more than one basic group B^+ in which case the diagram might be represented by $^-A)$—(B+ aspartic acid or $^-A)$—(B+ lysine

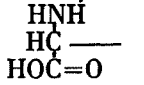


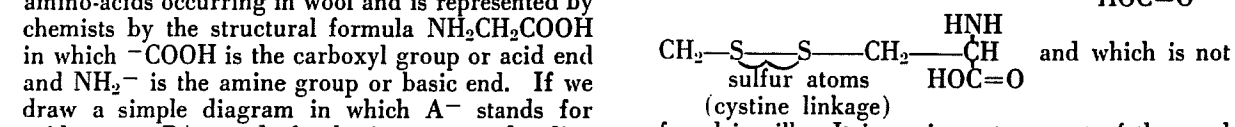
so now if two amino-acid chain structures happen to be adjacent lengthwise laterally to permit chemical union of the A^-s and B^+s as in the neutralization picture above, we can get a relationship something like the overly simplified imaginary diagram of electronic attraction of a salt bridge that follows:

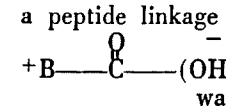


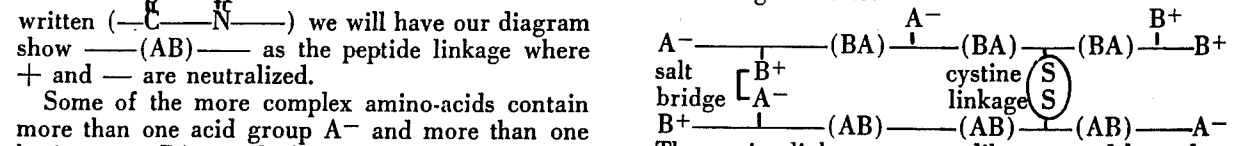
In this sketch  and  represent salt bridges and cross tie forces which attract H and ^-OH (water and acids and bases.) The unoccupied  or  site can accept an acid and the or  site a base.

4. How Do Wool and Hair Fibers Differ Chemically From Silk?

Wool contains a special amino-acid called cystine which can be shown graphically thus: 



and which is not found in silk. It is an important part of the wool protein structure, however, because it crosslinks neighboring peptide chains into a physically strong cross braced construction. In our simplified drawings the above formula may be represented  . When fitted into a large complex wool protein molecule we might find it appearing something like this:



The cystine linkages are readily ruptured by reduction. If very mild reducing agents are used, the link may be severed temporarily, at which time the wool or hair becomes very flabby and easily deformed. For instance, it can be curled or waved. The S——S bond may then be restored by mild and careful oxidation at which time the curl becomes set. This is the principle of the "home" permanent wave. In the case of dead or old wool, however, the reversal is incomplete and there is an appreciable loss of strength.

This link is quite sensitive to oxidation and reversing the permanent wave procedure by bleaching followed by reduction is also quite damaging. For this reason the conservator should avoid using bleaches especially on old wool because the control of bleaching is difficult and total destruction is likely.

The cystine linkage (sulfur to sulfur bond) is the substance relished by moths and carpet beetles since their digestive systems apparently demand it. Because silk does not contain this S——S bond, it is unpalatable to the insect and is not attacked. Insects will gnaw through silk, however, if it bars the way to wool.

In the aging process of wool, the S——S bond accepts oxygen readily and in doing so changes

from the disulfide insect foodstuffs to indigestible sulfur-oxygen substances. This explains why very old wool may not be relished by the moths.

5. Why Is It That New Silk Has High Tensile Strength Although It Lacks the Cystine Crosslinkage?

This is probably due to the way silk is formed when the silkworm spins its cocoon. The silk substance is extruded under tension which causes the molecules to be oriented lineally and packed close together. A resembling extrusion and stretching technique has been found to add great strength to modern synthetic fibers probably because of strong attraction forces where similar molecules are aligned very near to one another.

Wool is formed with the molecules more at random, and for that reason they are less closely aligned and more likely to contain wider spacings between and thus wool is weaker than silk.

6. Recognition Tests

Whenever a conservator or curator may want to establish the identity of a fiber he may choose between a number of different ways of testing. Recognition means which may enable us to recognize and distinguish between the several classes and subclasses are: Burning Tests; Visual Inspection; Magnification; Optical Microscopy; Electron Microscopy; X-ray Diffraction; Infra Red Spectroscopy; Ultra Violet Microscopy; Chemical and Stain Tests.

(1) *Burning Tests:* Perhaps the simplest test for chemical classification is the burning test. Cellulose fibers burn rapidly with a bright flame and produce a soft ash. When extinguished, the smoke odor resembles that of burning paper. Protein fibers burn more slowly producing a bead and the smoke odor is that of burning feathers. This test assumes that a small quantity of the unknown material can be spared for total destruction.

(2) *Visual Inspection:* A great many people can distinguish visually between new or undamaged materials. For this we rely on yarn luster, color, feel, drape and our estimate of the fiber size as we see it. None of these characteristics are completely trustworthy on old, worn or damaged fabrics. Luster is a light reflective property which can be changed by erosion, wear or accidental chemical action. The color of a fiber can change with ageing, light action, dyeings or accidental staining. The hand and draping qualities of a fabric can be drastically changed by wear, ageing and cleaning. Visual inspection does not involve destruction of the sample and so may be attractive from that point of view.

(3) *Examination by Magnifying Glasses:* This technique has the same advantages of permitting studies of the specimen without destruction of any part. However the usual practical reflective microscope or magnifying glass does not provide more than fifty power magnification. This is too low to permit positive differentiation between fine linen and silk, silk and some synthetic fibers, sheep's wool and some of the goat-camel hair or other animal fibers in case such fine distinction is desirable.

(4) *Optical Microscopy by Transmitted Light:* A more accurate and trustworthy method of visual analysis is by examination under transmitted light with 150 to 250 power magnification or greater. This entails mounting of a small sample on a glass slide and loss of that portion of the original. Since it is usually possible to spare a small section of the ends of one or two yarns, this loss may be permitted. Each of the four fiber subclasses or groups has its characteristic longitudinal and cross-sectional appearance.

(a) For example, new wool is generally cylindrical in shape but not quite a true circle when viewed endwise. The surface lengthwise is marked by irregular scales. With coarse hair, the scales are large and prominent, while very fine hairs or wools may be faintly marked. The appearance of the scales varies from one animal species to another and, to distinguish between these, the student must compare the appearance to known standards and sometimes resort to reactions of the slide samples to special stains or chemicals for positive confirmation. Figure No. 1 is an idealized sketch of a hair-wool fiber type. The rough surface as shown accounts for the lack of bright, reflective luster in new wool. In worn, aged or chemically damaged wool, however, the scales may disappear and the fabric becomes shiny as on an elbow of a used jacket.

(b) Two main types of silk may be encountered: wild and cultivated. As produced by the silkworm, the fibrous material in both consists of two different protein substances, a center material of twin filaments of exceedingly long, strong structure called *fibroin* cemented together by a glue-like physically weaker protein, *serecin.* The diet of the silk worm affects serecin profoundly. The wild silkworm feeds on leaves of trees containing a high percentage of tannin, a substance which renders protein difficultly soluble. Wild silk (Figure No. 2) thus is usually made up of the whole extruded substance. It is frequently tan colored, hard to bleach white, does not accept dyes readily, and is somewhat rough in texture. Often the fiber length (staple) is short because the cocoons are not gathered before the emerging moth splits them open.

The cultivated silk (Figure No. 3) on the other hand, is fed on mulberry tree leaves which are low in tannin content. In the "raw" state the serecin can be removed by hot alkaline detergent solutions and the two *fibroin* filaments can be readily cleaned. In this case the silk worm is killed before the moth develops and the whole extruded complex unwound from the cocoon prior to disoslving the serecin envelope.

The fibroin components are very fine fibers with the molecules apparently oriented chiefly in a

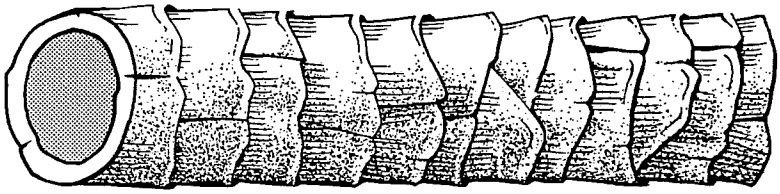


Fig. 1. *Idealized wool fiber*

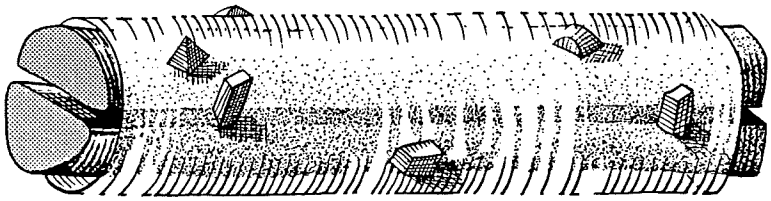


Fig. 2. *Idealized microscopic appearance of wild silk fiber*

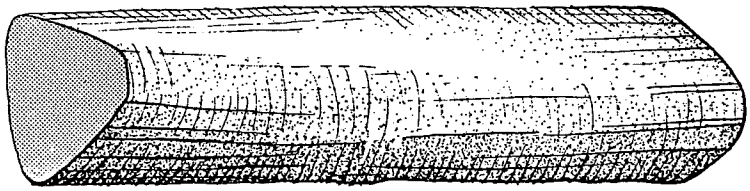


Fig. 3. *Idealized microscopic appearance of cultivated and "boiled off" silk*

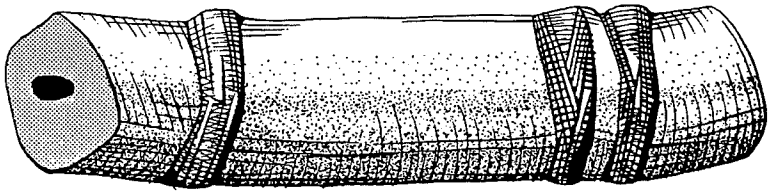


Fig. 4. *Idealized flax fiber*

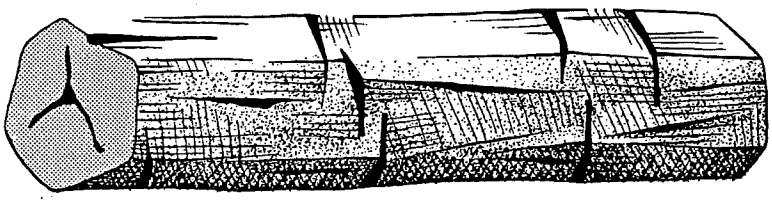


Fig. 5. *Idealized hemp fiber*

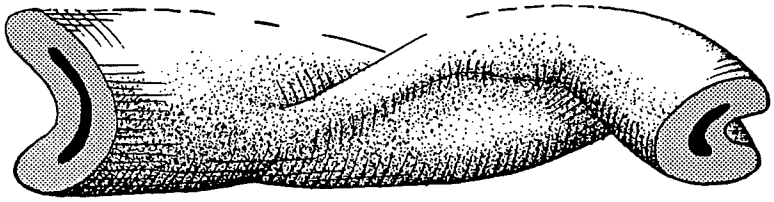


Fig. 6. *Idealized cotton fiber*

lengthwise direction. The structural cross section is irregularly polygonal in shape, but reasonably uniform in area along the entire length of the fiber which may reach 400 yards. Striations resembling gentle folds run along the lengthwise direction. These do not affect the high luster or light reflection properties. Damaged or badly worn silk may lose its characteristic luster and exhibit a surface fuzziness because of fraying out of fibrilles. This condition in relatively new silk is called "lousiness." Old silk is apt to be quite brittle and weak and may powder away in conservation treatments.

(c) Flax fiber (Figure No. 4) which when refined, spun into yarn and woven into fabrics, is called linen, is an ancient textile fiber which superficially might be mistaken for silk. It is one of the numerous bast type cellulosic fibers that may be encountered by the textile conservator. In the best grades of flax, individual fibers are small in cross section which is irregularly polygonal in shape and moderately long and smooth. Under transmitted light magnification it is marked by minute lengthwise striations and crosswise cell junctions or nodes slightly resembling a bamboo joint. Also a fine lumen or central channel may be detected. To emphasize these two characteristics, it may be necessary to stain the flax with methyl violet or chloride of zinc for confirmation.

The natural color ranges from pale yellowish white to pearl gray but this can be bleached white and may become quite lustrous.

Among other bast fibers of antiquity and primitive cultures, we sometime find hemp (Figure No. 5). This fiber resmebles flax in its random polygonal cross section. Also under the microscope, one may find cross shadow or transverse marking effects resembling the nodes of the flax. These are not so pronounced and the lumen or central channel is smaller in proportion and less easily seen. Hemp is generally a much coarser fiber than flax.

Jute is another bast fiber which superficially resembles flax in macro appearance. It is of little importance as museum specimen fiber except in modern carpets where it is found in the foundation structures. It differs from flax and hemp in that it is formed in part of ligno-cellulose, a substance which is slightly acidic and will accept basic dyes. Since it does not resist ageing, it will probably not appear in older fabrics.

(d) Cotton, a seed hair fiber is almost pure cellulose, (Figure No. 6). When mature, it is very small in cross sectional structure and ranges in length from about one-half to two or more inches, depending on the grade. Under a microscope, the cross sectional shape resembles a flattened or collapsed and twisted tube, the central space of which is the fiber lumen. Occasionally, the fibers are folded or bent.

The characteristic structure of cotton will swell and change to a more cylindrical shape when treated with strong alkalies or other chemicals. This is a peculiarity of chemically altered cotton such as mercerized fiber.

The color of the modern cultivated product is faintly gray. This responds readily to bleaching. It is possible that some primitive cottons may have been colored naturally, perhaps brown, although cellulose turns brown on wetting and oxidation anyhow, and must be bleached to return it to whiteness.

(5) Ultra violet reflection apparatuses provide another technique of studying a textile for identification purposes without destruction of the substance. The light change effects upon which this depends, however, are difficult to recognize and assess, especially if the fiber has ever been bleached, so that a great deal of practice in comparing unknowns against a standard is necessary before an individual can be confident in his judgment. Ultra violet microscopy provides means of identification by special wave length absorbtion phenomena similar to infra red spectrophotometry.

(6) The electron microscope is an ultra modern way of studying and identifying fibers. The equipment is quite costly and its use demands a highly trained scientist to operate and interpret the results. The same comments apply to X-ray photography.

(7) Infra red spectrophotometry, a very recent analysis technique promises a way not only of identifying the fiber content but also of studying any changes that may have come about. A small quantity of the specimen must be sacrificed. Like other very modern analytical tools, the instrument is costly and a great deal of study and experience in interpreting the results is necessary.

(8) Most chemical fiber identification tests involve destruction of the sample. For that reason we may be reluctant to proceed. A few such tests follow. In general, hot strong alkali (potassium hydroxide 5%) solutions will dissolve protein fibers but will not appreciably affect cellulosics. (However, jute may be disintegrated by hot alkalies.) Hot 5 to 10% solutions of sulfuric acid will dissolve cellulose but will not affect proteins appreciably. Hot 5% hydrocloric acid will destroy silk but not wool. Dilute nitric acid stains silk and wool yellow but will not stain flax, cotton or synthetic fibers. Zinc-chlor-iodide reagent stains flax brownish violet and hemp true violet. Cotton is colored red to violet with it, whereas wild silk is yellow. Millon's Reagent, a freshly prepared mercuric nitrate, gives a brick red color with proteins but no color with cellulose. All of these require an appreciable amount of the material in question and even then we may not be able to distinguish between closely related fibers. For example, it takes much experience and practice to differentiate between the various wools and camel-goat hair fibers where one must depend on minute differences in scale appearance and pigmentation. This presupposes accurate and trustworthy comparison standards which may not be available.

A summarization of other characteristics and behavior patterns is included with this article: "Tabulations of Recognition Characteristics for the Most Common Ancient Fibers," which we hope will be helpful in future studies, especially in connection with cleaning problems.

TABULATIONS OF RECOGNITION CHARACTERISTICS FOR THE MOST COMMON ANCIENT FIBERS

PROPERTY	ADDED COMMENTS	WOOL	SILK (Domesticated)	FLAX OR LINEN	COTTON
1. Source		animal	animal	vegetable	vegetable
2. Chemical Classification		protein	protein	cellulosic	cellulosic
3. Other Classification		hair	insect secretion	bast	seed hair
4. Resembling Fibers		alpaca, goat, camel	spider web, etc.	hemp, ramie, leaf	kapok, paper
5. Appearance	a. color (not dependable)	varies: white, brown, gray, black	gray to canary yellow	gray to tan	white to faintly gray
	b. luster (may change)	non-lustrous when new	highly reflective	may be quite lustrous	non-reflective
	c. diameter (not specific)	varies greatly	fine, uniform	fine to coarse	fine, fairly uniform
	d. length (not specific)	moderately short	very long	moderately long	short
6. Burning Test	a. burning rate	slow	moderate	fast	fast
	b. odor	like feathers	like feathers	like paper	like paper
	c. ash	brittle bead	brittle skeleton	soft, powdery	soft, powdery
7. Transmitted Light Microscopy	a. outstanding characteristic	outer cuticle with scales	smooth, slightly striated, no skin or outer covering	lengthwise striations, crosswise joints, small center lumen	flat twisted tube with flat lumen
	b. illustration	see Figure 1	see Figures 2 and 3	see Figures 4 and 5	see Figure 6
8. Other Practical and Simple Identification Tests	a. Millon's Reagent	red color	red color	no red color	no red color
	b. 5% warm K O H	dissolves	dissolves	no change	dissolves a little
	c. 5% warm sulfuric acid	no change	no change	dissolves	dissolves
9. Damaging Chemical Effects	a. strong alkalies	will dissolve wool	will dissolve silk	not serious, depends on history	little to not serious, depends on previous history
	b. weak alkalies	will felt wool if worked or heated	no appreciable effect	very little, depends on previous history	very little or none, depends on previous history
	c. strong acids	wool resists	silk is dissolved or damaged	dissolves flax	dissolves cotton
	d. weak acids	not affected	not affected, except by H C l	very little	very little
	e. oxidizing bleaches	will weaken or damage	will weaken or damage	slightly affected	will withstand when new
	f. reducing bleaches	will weaken	no serious effect	no appreciable effect	no appreciable effect
	g. salt	no effect	damages silk	no effect	no effect
10. Other Destructive Effects	a. light	wool sunburns and weakens	very easily damaged	same as with cotton	tendering results
	b. rain wetting	promotes ozone and fungi damage	promotes ozone, acid and salt damage	same as for cotton	promotes acid, ozone and fungi damage
	c. atmospheric fumes	not sensitive to acid fumes, but may be to ozone	slightly sensitive to acid fumes. Beware of salt spray	same as for cotton	acid fumes cause tendering
	d. insects	beware of moths and scavenger beetles	not so apt to be attacked by insects	same as for cotton	silverfish and starch-loving beetles devour it
	e. fungi	resists attack more than cotton	resists attack	easily attacked	very easily attacked